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INTERLABORATORY STUDY 91-2

N-NITROSODIMETHYLAMINE IN EFFLUENTS AND GROUNDWATER

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INTERLABORATORY STUDY 91-2

N-NITROSODIMETHYLAMINE IN EFFLUENTS AND GROUNDWATER

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1 SUMMARY OF INTERLABORATORY STUDY 91-2

Interlaboratory Study 91-2 was initiated as part of an on-going program of laboratory performance management studies conducted by the Quality Management Office of Laboratory Services Branch, Environment Ontario. It assesses the performance of participating laboratories for the analysis of N-nitrosodimethylamine (NDMA) in spiked effluents and in groundwater. Thirteen laboratories (government, industry, and commercial) agreed to participate in this study. Results were received from eleven participants.

The results demonstrate that the major source of variation among the participants is a difference of standards. Results from the ampouled standards provided as part of this study showed considerable differences, high and low, from the design values.

Performance among the laboratories for the spiked matrix samples demonstrated good within-laboratory precision for one of the effluents and the groundwater sample. Results from the other effluent were more variable, possibly indicating some matrix effects. The variability in the results for the spiked effluents may also be attributed to inappropriate spiking levels of d_6-NDMA. The importance of a 1:1 ratio of native to deuterated NDMA has been noted in previous interlaboratory studies[1,2], and this study continues to support this need in the analytical method.

The results from this study do not show a significant difference in the performance of laboratories using High Resolution Mass Spectrometry (HRMS) than from those participants using Low Resolution Mass Spectrometry (LRMS).

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards. The Quality Management Office, Laboratory Services Branch, Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability among laboratories for the analysis of N-nitrosodimethylamine (NDMA) in spiked effluents and unspiked contaminated groundwater. This study was designed to expand the assessment of laboratories analyzing for NDMA beyond the group of laboratories participating in previous interlaboratory studies[1,2] for this parameter.

Thirteen laboratories (government, industry, and commercial) agreed to participate in this study. Results were received from eleven participants. A list of participants is given in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Samples

The stock spiking solution was prepared from a concentrated NDMA solution of 97 µg/mL NDMA in ethanol. This solution, certified by the supplier, is traceable to USDA Reference 7 Nitrosamine Standard, was obtained from Thermedics Inc., Woburn MA, Lot #322-69. A spiking solution for the matrix samples was prepared by diluting the concentrated stock solution in methanol. The volumetric flask containing the dilute solution was stored overnight in a freezer at -20°C. The solution was sealed into 5 mL amber ampoules the following day. The ampoules were stored at 4°C in the dark.

Fifty litres of two different Sewage Treatment Plant (STP) effluents were collected on April 11, 1991 from the Kitchener STP and the Brantford STP. A 35 litre sample of contaminated groundwater was also collected on the same day from a test well near Elmira, Ontario. Each separate sample matrix was homogenized in stainless steel tanks. Particulate matter was allowed to settle out of each bulk matrix sample. The sample was then decanted into another stainless steel tank and stirred for 15-20 minutes to homogenize the sample. The bulk samples were then dispensed into 1 litre amber bottles, filling the bottles (mean volume was 1040 mL ±2 mL).

The Kitchener STP effluent was labelled the "1" series, the Brantford STP effluent was labelled the "2" series, and the groundwater was labelled the "3" series. The groundwater samples were not spiked with the NDMA spiking solution. These samples were labelled "3A" or "3B" and the participants received one of each. The two STP effluents were spiked at two different levels. The unspiked samples were labelled "1A" or "2A". The low spikes were labelled "1B" or "2B". The high spikes were labelled "1C" or "2C". All spiking of individual bottles was done using a repeating microlitre syringe. All design values are given in Table 1, Appendix 1.

The ampouled standards for direct instrumental analysis were serially diluted from a concentrated stock solution. The stock solution was prepared from the following neat compounds: NDMA from Supelco Inc., Lot #LA03531 and d_6-NDMA from MSD Isotopes, Lot 4002-0. The stock solution was prepared in methanol and hand-sealed into 5 mL amber ampoules. Subsequent dilutions were prepared in dichloromethane and hand-sealed into 5 mL amber ampoules. The ampoules submitted to the participants were labelled NDMA STD1 and NDMA STD2. Concentrations are given in Table 2, Appendix 1.

3.2 Sample Distribution

Samples were cooled and then packed in cartons lined with Styrofoam chips. The ampoules were placed in Stryofoam holders and inserted into a sample box. The samples to the U.S. participant were shipped on April 30, 1992. Samples to the remaining participants were shipped via Purolator Courier on May 1, 1991. A list of the laboratories receiving sample sets is given in Appendix 2. A copy of all correspondence is also included in Appendix 2. Participants were requested on the report form provided (Appendix 2) to summarize their Sample Preparation Principles, Instrumental Measurement Method Principles, and indicate the Gas Chromatographic Column used for the NDMA analysis. Participants were also asked to indicate the extraction volume, injection volume, and level of d_6-NDMA spiked into the samples. All participants were assigned a unique identification code. Participants were also advised by telephone after the samples had been distributed, of the approximate ranges of the spiked samples, as there was concern from several participants that unknown high levels may cause instrument problems.

Laboratory 4010 had problems with their initial extraction of the samples. They were provided with a replacement set on May 14, 1991.

3.3 Data Evaluation Technique

Results were submitted to the Quality Management Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet.

The participating laboratories who reported results were mailed a copy of the tables of results on July 5, 1991 and asked to confirm their results. No corrections were made to the data set.

The mean, median and standard deviation were calculated for each parameter in each sample and are included in Table 1, Appendix 1. Results of the spiked NDMA samples were converted to percent recovery of the target value and are given in Table 3, Appendix 1. Results from the ampouled standards provided for direct instrumental analysis are given in Table 2, Appendix 1.

The results for the spiked samples (1B, 1C, 2B, and 2C), converted to percent recovery of the design value, were plotted using the Youden technique[3]. The Low Spikes (1B and 2B) were plotted as one Youden pair (Figure 1) and the High Spikes (1C and 2C) were plotted as a second Youden pair (Figure 2). The results for the duplicate groundwater samples (3A and 3B) were also plotted using the Youden technique (Figure 3). All graphs are in Appendix 1.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE

All the participants but one (Laboratory 4011) were able to detect NDMA in the spiked and unspiked effluent samples (1A-C and 2A-C). The variability among the samples appeared consistent between the two effluents, as the Standard Deviations of the unspiked samples were very similar, the Standard Deviations of the Low Spikes (1B and 2B) were very similar, and the Standard Deviations of the High Spikes (1C and 2C) were very similar (Table 1).

When corrected for the background level of NDMA in the effluents (1A and 2A), the interlaboratory mean and median for the Low Spikes (1B and 2B) are higher than the target value by 15-20%. The interlaboratory mean and median of the High Spikes (1C and 2C) show good agreement with the target value. Most participants used a d_6-NDMA spike level that was closer to the High Spike level than to the Low Spike level. This results in a closer 1:1 relationship between the native and deuterated NDMA in the samples and greater accuracy is achieved when quantitating the results. The importance of the 1:1 relationship between the native and deuterated NDMA for achieving greater accuracy has been noted in previous interlaboratory studies[1,2]. The effect of greater accuracy for the High

Spike is demonstrated in the Youden plots for the two effluent samples (Figures 1 and 2). The results for the High Spikes (1C and 2C) cluster tightly around the target with a minimal spread along the line drawn between the origin and the target (Figure 2). The results for the Low Spikes (1B and 2B) show more systematic bias between the participants, as the results are spread out along the line drawn between the origin and target (Figure 1).

The most common source of variability among the participants appears to be a difference of standards. The results for the two ampouled standards provided for direct instrumental analysis (STD1 and STD2) show considerable variation among the participants (Table 4). All the participants used the same injection volume (2 µL). The high or low biases do not appear related to different instrument detection systems (Low Resolution Mass Spectrometry (LRMS) or High Resolution Mass Spectrometry (HRMS)), as high and low biased results are observed for both instruments. This difference in standards will contribute to the variation among the laboratories for the results of the spiked effluent samples, as well as the differences in the level of the internal standard (d_6-NDMA), as noted above.

The results for the duplicate groundwater samples (3A and 3B) indicate that most laboratories were able to demonstrate reasonable within-laboratory precision. When plotted using the Youden technique (Figure 3), the results fall on a 45° line that passes through the origin and the interlaboratory mean and/or median, demonstrating the good within-laboratory precision. However the participants' results are spread along this line, indicating between-laboratory bias. Once source of bias, is a difference in standards, as noted above. Another possible source of bias may be related to matrix effects that a laboratory's method may not be suited for. This groundwater sample was chosen from a site known to be contaminated with Chlorobenzene (Dr. Vince Taguchi, MOE, LSB and Gord Robinson, MOE, West Central Region, personal communication). Chlorobenzene elutes with the same retention time as native NDMA when using a DB-1701 Gas Chromatographic column. (Deuterated NDMA elutes 1 second earlier on the DB-1701 column.) The Chlorobenzene therefore may interfere with the accurate quantitation of NDMA. Laboratories that were biased high may have had insufficient resolution between the Chlorobenzene and NDMA peaks, giving a high result.

INDIVIDUAL LABORATORY PERFORMANCE

Laboratory 4001

This laboratory's results for the ampouled standards (STD1 and STD2) were low relative to the target values, though they had good agreement with the interlaboratory mean and median. Their results for the spiked effluent samples (1B, 1C, 2B and 2C) were biased high, relative to the design value. This bias in the samples may be attributed to their low standards. Their results for the groundwater samples (3A and 3B) had very good agreement with the interlaboratory mean and median. They demonstrated good within-laboratory precision.

Laboratory 4002

Laboratory 4002's results for the ampouled standards (STD1 and STD2) were a little low relative to the target values. They were also biased low for the spiked effluent samples (1B, 1C, 2B and 2C). Their results for the groundwater samples (3A and 3B) were below the interlaboratory mean and median. They demonstrated good within-laboratory precision.

Laboratory 4003

Laboratory 4003 noted with their results that the extractions had been completed prior to being advised of the approximate ranges of the samples, so the spike level of d_6-NDMA may not have been appropriate. They were biased low for the ampouled standards (STD1 and STD2), relative to the target values and the interlaboratory mean and median. They were biased slightly high for the spiked effluent samples (1B, 1C, 2B and 2C). This high bias may be due to a difference in their standards or to non-ideal addition of d_6-NDMA, as noted above. Laboratory 4003's results for the groundwater samples (3A and 3B) were very low compared to the other participants. They had noted that their results for these samples had been quantitated using an external standard calculation and had not been corrected for surrogate recovery. This may be the source of their low bias for these samples.

Laboratory 4005

This laboratory's results for the ampouled standards (STD1 and STD2) were biased low relative to the target values. Their results for the spiked effluent samples (1B, 1C, 2B and 2C) were also biased low. Their results for the groundwater samples (3A and 3B) were below the interlaboratory mean and median. They demonstrated good within-laboratory precision.

Laboratory 4006

Laboratory 4006 had low results for STD1 and high results for STD2, relative to the design values. This suggests curvature of their calibration range. Their results for the spiked effluent samples (1B, 1C, 2B and 2C) were biased high compared to the design value and to the interlaboratory mean and median. They were also biased high for the groundwater samples (3A and 3B).

Laboratory 4008

Laboratory 4008 noted with their results that they had had extraction problems with sample 2A and were unable to report any results for that sample. They had good agreement with the target values for STD1 and was slightly low for STD2. They were biased high for the lower spiked effluent samples (1B and 2B) and had excellent recovery for the higher spiked effluent samples (1C and 2C), relative to the design values. These results suggest curvature at the lower end of their calibration range. This resulted in variable within-laboratory precision, particularly for the Low Spikes (Figure 1). However they had excellent within-laboratory precision for the groundwater samples (3A and 3B) and good agreement with the interlaboratory mean and median.

Laboratory 4009 and 4009B

This laboratory analyzed the samples using two different detection systems, LRMS (4009 results) and a Thermal Energy Analyzer (TEA) (4009B results). They did not detect the NDMA in the low standard (STD1) using LRMS and were biased low for the high standard (STD2). Their results for the Low Spikes (1B and 2B) were slightly lower than the target values (Figure 1). However they had excellent agreement with the design values for the High Spike effluent samples (1C and 2C). This suggests some curvature at the lower end of the calibration range and possibly insufficient sensitivity at this end. They were biased high relative to the interlaboratory mean and median for the groundwater samples (3A and 3B). They demonstrated good within-laboratory precision for all samples.

The TEA system (4009B) does not distinguish between native and deuterated NDMA. Their total result for STD2 is somewhat high compared to the total target value of native and deuterated NDMA in STD2. Their total result in STD1 is low compared to the total target value of native and deuterated NDMA. This suggests some curvature in their calibration curve. They had good agreement with the design values for all the spiked effluent samples (1B, 1C, 2B and 2C). They were biased high relative to the interlaboratory mean and median for the groundwater samples (3A and 3B). They demonstrated good within-laboratory precision for all samples.

Laboratory 4010

As noted in Section 3.2, this laboratory had problems with their initial extraction of the samples and they received a replacement set of samples several days later. They had excellent agreement with the target values for the ampouled standards (STD1 and STD2). Their results for the spiked effluent samples were variable, possibly due to the later date in extracting the second set of samples. Their results for the lower spikes (1B and 2B) were biased high relative to the design value (Figure 1), while the high spikes (1C and 2C) were slightly low relative to the design value (Figure 2). However, they had excellent within-laboratory precision for the groundwater samples (3A and 3B) and had the interlaboratory median results. This suggests some inconsistency in applying their method, which should easily be improved.

Laboratory 4011

Laboratory 4011 did not detect any NDMA in the ampouled standards, nor in the spiked effluent samples (1B, 1C, 2B, and 2C). Their results for the groundwater samples (3A and 3B) were very low compared to the interlaboratory mean and median. This laboratory did not spike the samples with d_6-NDMA. These results suggest that their method is not suitable for the analysis of the types of samples submitted in this study. The non-detection of NDMA in the ampouled standards appears to indicate that their detection system is not calibrated appropriately for the analysis of this compound.

Laboratory 4012

Laboratory 4012 reported that they had various problems with their procedures during the time of this interlaboratory study. Software modifications to their HRMS resulted in downtime that delayed the analysis of the sample extracts. The initial sample extracts were also lost and extraction had to be repeated on smaller aliquots of sample and minus an acid wash step that had been applied to the first set of extractions. Several of the extracts were also analyzed on a LRMS instrument to provide support data for the results reported from the modified HRMS. These results have been labelled 4012A and 4012B. The LRMS data was excluded from the statistical evaluations except for the set of results for samples 3A and 3B, as a full set of results was not reported for the other samples and the standards.

The HRMS results for the ampouled standards (STD1 and STD2) were biased high relative to the design values. Their LRMS results showed better agreement with the design values. This suggests that further comparisons between the two analytical systems was required. The results for the spiked effluent samples (1B, 1C, 2B and 2C) are variable. The recovery for sample 1B was higher than the design value but the recoveries for samples 1C, 2B, and 2C were lower than the design value. The results for the groundwater samples (3A and 3B) were high compared to the interlaboratory mean and median, with better within-laboratory precision achieved by the LRMS than the HRMS. The various problems noted by the participant above most likely caused the variability in their results for this interlaboratory study. Improvements to their procedures should improve their performance.

Laboratory 4013

Laboratory 4013's results for the ampouled standards showed good agreement with the target values. Their recoveries for the lower spiked effluents (1B and 2B) were high relative to the design values (Figure 1), while their recoveries for the higher spiked effluents (1C and 2C) were slightly lower than the design values (Figure 2). Their within-laboratory precision was also better for the High Spikes than the Low Spikes. Their within-laboratory precision was good for the groundwater samples (3A And 3B) and are close to the interlaboratory mean and median.

5 REFERENCES

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6 APPENDIX 1: TABLES AND GRAPHS

Table 1 Effluent and Groundwater Samples; Results in µg/L

Table 2 Spiked Effluent Results Corrected for Background; Results in µg/L

Table 3 Spiked Effluent Sample Results Converted to Percent Recovery of Design Value (Background Corrected)

Table 4 NDMA and d_6-NDMA Standard Results in pg/µL

Figure 1 Youden Plot of Spiked Effluent 1 Samples (Percent Recovery)

Figure 2 Youden Plot of Spiked Effluent 2 Samples (Percent Recovery)

Figure 3 Youden Plot of Groundwater Samples (µg/L)

TABLE 1 - EFFLUENT AND GROUNDWATER SAMPLES
RESULTS IN μg/L

CODE/SAMPLE	1A	1B	1C	2A	2B	2C	3A	3B	INSTRUMENT
SOURCE	KITCHENER STP			BRANTFORD STP			GROUNDWATER		-
SPIKE	-	0.302	0.588	-	0.302	0.588	-	-	-
4001	0.042	0.451	0.756	0.05	0.422	0.779	670	667	LRMS
4002	0.068	0.298	0.552	0.059	0.305	0.477	518.7	563.6	LRMS
4003	0.104	0.488	0.794	0.093	0.549	0.814	291	270	LRMS
4005	0.10	0.30	0.70	0.088	0.30	0.59	590	580	HRMS
4006	0.124	0.495	1.11	0.109	0.539	1.00	825	802	HRMS
4008	0.104	0.595	0.702	N/A **	0.384	0.632	677	662	LRMS
4009	0.072	0.362	0.705	0.086	0.365	0.687	765	841	LRMS
4009B	0.078	0.410	0.739	0.089	0.393	0.698	759	811	TEA
4010	<0.010	0.519	0.527	<0.01	0.505	0.498	683	695	LRMS
4011	ND	ND	ND	ND	ND	ND	270	315	LRMS
4012	0.079	0.432	0.579	0.123	0.385	0.488	847	740	HRMS
4012A #		0.361	0.655				795*	731*	HRMS/LRMS*
4012B #								729	LRMS
4013	0.066 i	0.401	0.589	0.095 i	0.494	0.641	761	704	LRMS
MEAN	0.070	0.396	0.646	0.066	0.387	0.609	650.1	644.7	
MEDIAN	0.078	0.432	0.702	0.0885	0.393	0.641	683	695	
STD DEV	0.0340	0.0913	0.1613	0.0350	0.0880	0.1584	161.23	174.06	
n	11	11	11	10	11	11	15	15	

LEGEND: LRMS - Low Resolution Mass Spectrometer
HRMS - High Resolution Mass Spectrometer
TEA - Thermal Energy Analyzer

# Additional analyses provided by this participant. Only the original data was used in the evaluation (except for second set of results for samples 3A and 3B).

** Participant was unable to complete the analysis for this sample.

TABLE 2 - SPIKED EFFLUENT RESULTS CORRECTED FOR BACKGROUND RESULTS IN μg/L

SAMPLE	1B	1C	2B	2C
SPIKE	0.302	0.588	0.302	0.588
4001	0.409	0.714	0.372	0.729
4002	0.23	0.484	0.246	0.418
4003	0.384	0.690	0.456	0.721
4005	0.20	0.60	0.212	0.502
4006	0.371	0.986	0.43	0.891
4008	0.491	0.598	0.384	0.632
4009	0.29	0.633	0.279	0.601
4009B	0.332	0.661	0.304	0.609
4010	0.519	0.527	0.505	0.498
4012	0.353	0.500	0.262	0.365
4013	0.335	0.523	0.399	0.546
MEAN	0.356	0.629	0.350	0.592
MEDIAN	0.353	0.60	0.372	0.601
STD DEV	0.0971	0.1416	0.0951	0.1509

TABLE 3 - SPIKED EFFLUENT SAMPLE RESULTS CONVERTED TO PERCENT RECOVERY OF DESIGN VALUE (BACKGROUND CORRECTED)

SAMPLE	1B	1C	2B	2C
SPIKE	0.302	0.588	0.302	0.588
4001	135%	121%	123%	124%
4002	76%	82%	81%	71%
4003	127%	117%	151%	123%
4005	66%	102%	70%	85%
4006	123%	168%	142%	151%
4008	162%	102%	127%	107%
4009	96%	108%	92%	102%
4009B	110%	112%	101%	103%
4010	172%	90%	167%	85%
4012	117%	85%	87%	62%
4013	111%	89%	132%	93%
MEAN	117.7%	106.8%	115.7%	100.6%
MEDIAN	117%	102%	123%	102%
STD DEV	32.10%	24.06%	31.45%	25.64%

TABLE 4 - NDMA AND d_6-NDMA STANDARDS
RESULTS IN pg/µL

AMPOULE	STD1 - native	STD 1 - d6	STD2 - native	STD 2 - d6	INSTRUMENT
TARGET	25.27	26.04	252.7	260.4	
4001	19	18	160	195	LRMS
4002	22	22.4	231.4	237.7	LRMS
4003	8	9	143	137	LRMS
4005	15	15	150	160	HRMS
4006	16.1	17.1	262	298	HRMS
4008	23.5	24.4	226	232	LRMS
4009	ND	ND	121	109	LRMS
4009B	28	#	574	#	TEA
4010	26	24	248	259	LRMS
4011	ND	ND	ND	ND	LRMS
4012	38.4	37.2	294	337	HRMS
4012A *	27.5		243	273	LRMS
4012B *			301		LRMS
4013	24	23	244	254	LRMS
MEAN	17.5	17.3	189.0	201.7	
MEDIAN	19	18	226	232	
STD DEV	11.49	11.06	84.17	95.42	
n	11	11	11	11	

LEGEND:
LRMS - Low Resolution Mass Spectrometer
HRMS - High Resolution Mass Spectrometer
TEA - Thermal Energy Analyzer

* Additional analyses provided by this participant. Only the original data was used in the evaluation.

# TEA result is total of native and deuterated NDMA. It is not included in statistical calculations.

FIGURE 1: N-NITROSODIMETHYLAMINE

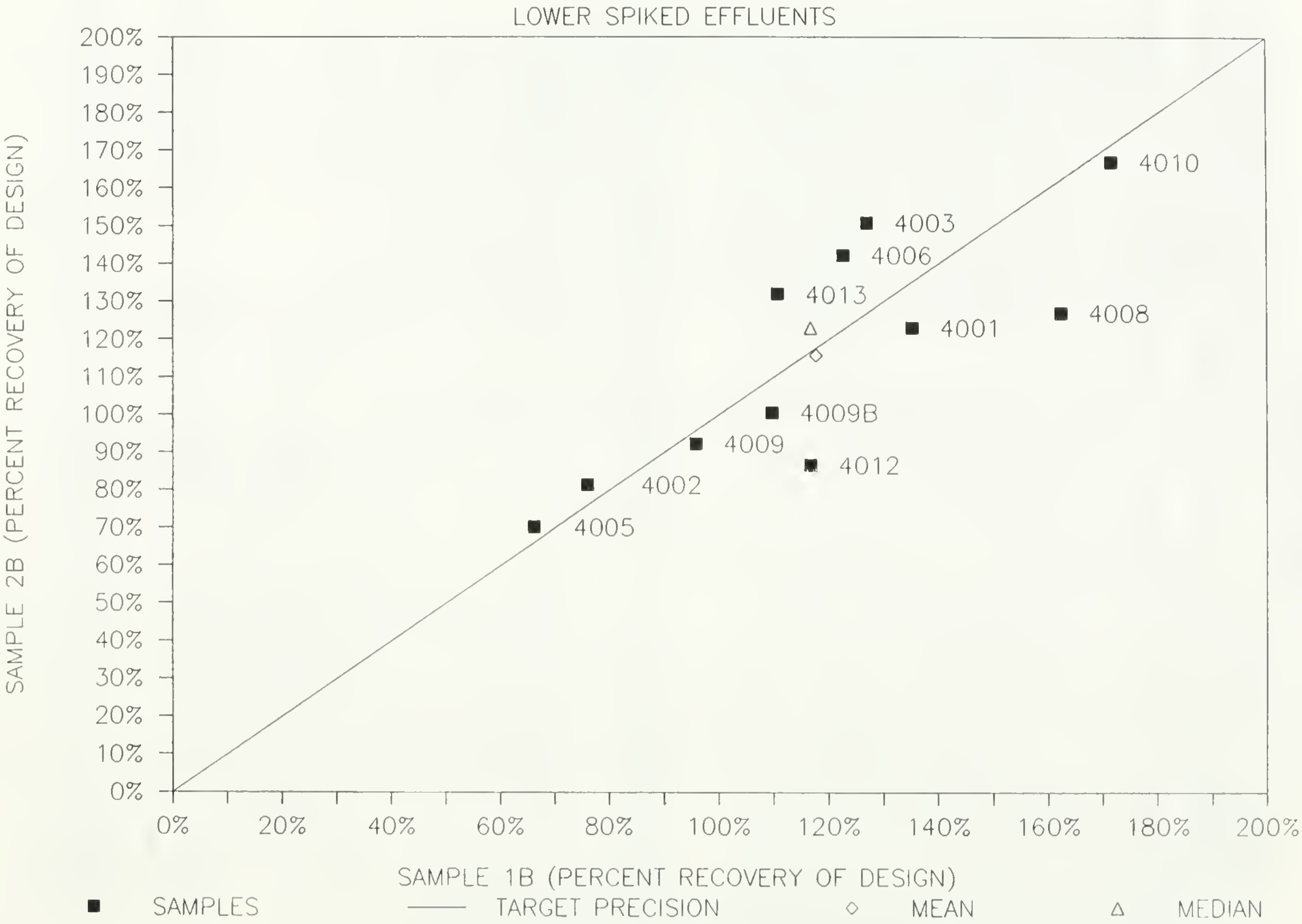


FIGURE 2: N-NITROSODIMETHYLAMINE

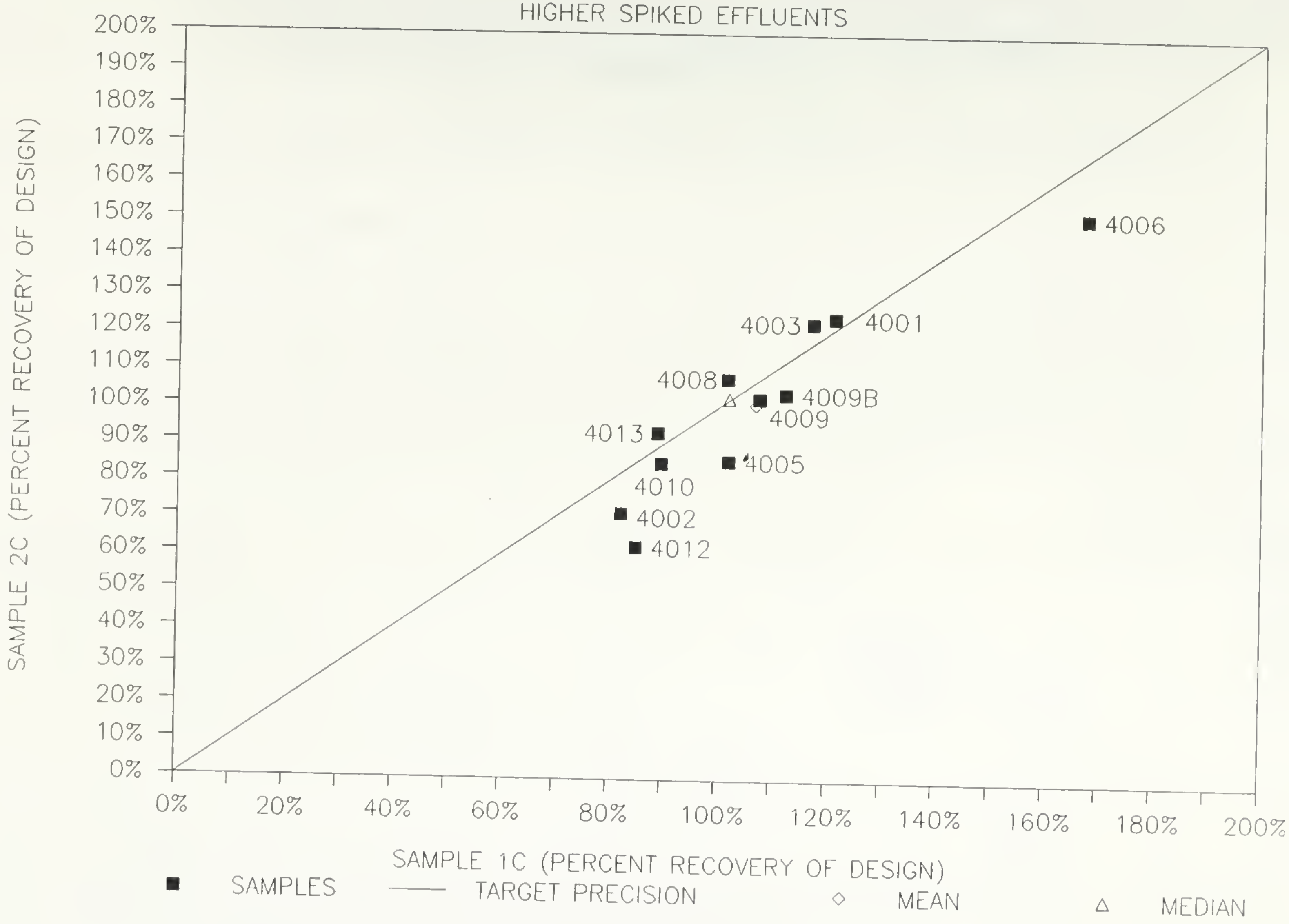
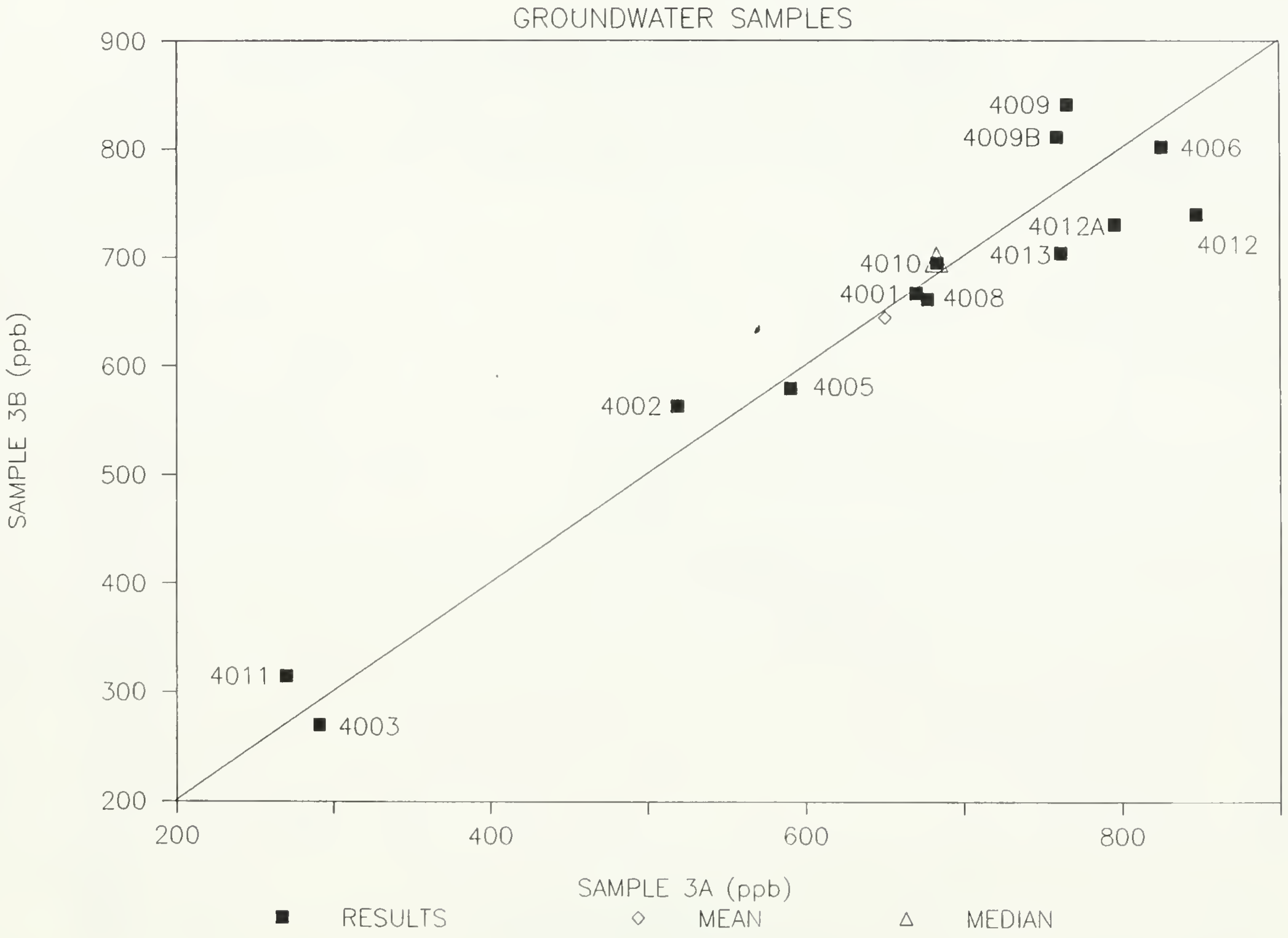


FIGURE 3: N–NITROSODIMETHYLAMINE


GROUNDWATER SAMPLES

7 APPENDIX 2: LIST OF PARTICIPANTS AND CORRESPONDENCE

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Contact: Annette Bibaud
(519) 747-2275

BAS Labs Ltd.
14 Abacus Road
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Contact: Daniel Andrews
(416) 458-4044

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G-20
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Contact: Al Nitowski
(203) 573-3495

Ontario Hydro
Research Division
800 Kipling Ave., KR310
Toronto, Ontario
M8Z 5S4

Contact: Ron Massey
(416) 231-4111 Ext. 7100

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF N-NITROSODIMETHYLAMINE

STUDY NO. 91-2

INTRODUCTION

Laboratories receiving this notification are invited by Environment Ontario, Laboratory Services Branch, to participate in an interlaboratory variability study of spiked effluents. Laboratories interested in participating in this program, scheduled for the week of April 29, 1991 should contact Sylvia Cussion at (416) 235-5842 of Environment Ontario to confirm participation. Written confirmation (attached response sheet) must be submitted to Environment Ontario no later than April 24, 1991 (FAX: (416) 235-5744).

STUDY DESIGN

This study is being conducted to assist laboratories in assessing their analytical performance. Sample sets will consist of three samples per matrix: one unspiked and two spiked effluent samples for a total of nine (9) samples, and two (2) ampoules containing native and deuterated N-Nitrosodimethylamine for direct instrumental analysis.

Parameter to be spiked into samples: N-Nitrosodimethylamine (CAS # 62-75-9)

Time Limit: 4 days storage at 4°C in the Dark prior to extraction.
Samples will not be preserved.

Ampoules are to be stored at 4°C in the Dark until ready for analysis. Time limit is the due date for Interlaboratory Study results.

SCHEDULE

During the week of April 29, 1991, participating laboratories will receive a total of nine (9) samples divided as follows: three (3) each of three different effluent samples plus two (2) ampoules for direct instrumental analysis. All samples will be for the analysis of N-Nitrosodimethylamine.

Participating laboratories are expected to initiate analysis of the samples within the time limits specified above. Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within thirty (30) days of receipt of the samples to Sylvia Cussion at the following address:

Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories reporting results will receive a complete set of the results where they will be identified only by the identification code. Recommendations made by Environment Ontario will be included in the final report. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this interlaboratory study (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF N-NITROSODIMETHYLAMINE

STUDY 91-2

	YES	NO
We will participate in MOE study 91-2 for the analysis of N-Nitrosodimethylamine.	☐	☐

Minimum amount of sample volume required: ____________________

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address:	Shipping Address:
____________________	____________________
____________________	____________________
____________________	____________________
____________________	____________________
____________________	____________________

Contact Person: ____

Telephone: ____

Please return this response form by April 24, 1991 to:

Sylvia Cussion
Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

(416) 235-5842
FAX: (416) 235-6110

April 30, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-2

Please find enclosed eight (8) 1000 mL amber glass bottles plus two (2) 5 mL amber ampoules. If you are missing any of the above samples or they have been broken in transit, please contact me at the above phone number immediately.

The eight samples are to be analyzed for N-Nitrosodimethylamine (NDMA). They are labelled as follows:

Effluent 1:	1A	1B	1C
Effluent 2:	2A	2B	2C
Effluent 3:	3A	3B	

Analysis of the samples must be initiated within four days of receipt of samples. Store all samples in a refrigerator at 4 ±2 degrees Celcius **in the dark** until ready for analysis. A report form is attached.

The ampoules are pure NDMA standards containing native and deuterated NDMA in dichloromethane. They are to be analyzed by direct injection and the results reported on the attached form. The ampoules are labelled as follows:

Ampoules:	NDMA STD 1	NDMA STD 2

Results of the eight samples and two ampoules are to be submitted by June 7, 1991. A report form is included with your lab identification number and the sample numbers described above.

Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842
FAX (416) 235-6110

April 30, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-2

Please find enclosed two sets of eight (8) 1000 mL amber glass bottles plus two (2) 5 mL amber ampoules. If you are missing any of the above samples or they have been broken in transit, please contact me at the above phone number immediately.

The eight samples are to be analyzed for N-Nitrosodimethylamine (NDMA). They are labelled as follows (in duplicate):

Effluent 1:	1A	1B	1C
Effluent 2:	2A	2B	2C
Effluent 3:	3A	3B	

Analysis of the samples must be initiated within four days of receipt of samples. Store all samples in a refrigerator at 4 ±2 degrees Celcius **in the dark** until ready for analysis. A report form is attached.

The ampoules are pure NDMA standards containing native and deuterated NDMA in dichloromethane. They are to be analyzed by direct injection and the results reported on the attached form. The ampoules are labelled as follows:

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Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842
FAX (416) 235-6110

INTERLABORATORY STUDY 91-2

REPORT FORM: N-NITROSODIMETHYLAMINE (NDMA)

REPORT DUE DATE: JUNE 7, 1991

BORATORY IDENTIFICATION CODE:

MPLE	RESULTS (g/L)	EXTRACTION VOLUME	FINAL VOLUME OF EXTRACT	INJECTION VOLUME	d_6 SPIKE LEVEL

MPLE PREPARATION PRINCIPLES:

STRUMENTAL MEASUREMENT METHOD PRINCIPLES:

S CHROMATOGRAPHIC COLUMN:

INTERLABORATORY STUDY 91-2

REPORT FORM: N-NITROSODIMETHYLAMINE (NDMA)

REPORT DUE DATE: JUNE 7, 1991

LABORATORY IDENTIFICATION CODE:

AMPOULE	RESULT: NATIVE (pg/ L)	DEUTERATED (pg/ L)	INJECTION VOLUME
NDMA STD 1			
NDMA STD 2			

Laboratory Services Branch
Quality Management Unit
125 Resources Rd.
Rexdale, Ontario
M9W 5L1

July 5, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-2

Thank you for your participation in Interlaboratory Study 91-2 for the analysis of N-Nitrosodimethylamine (NDMA) in Effluents. Please find enclosed a copy of the Tables of Results from all participants, the spiking level values for two of the effluents (the third effluent was not spiked), and the target values for the ampouled standards.

Please review the results for your laboratory for any transcription errors and report any corrections to me by no later than July 31, 1991. A final report will be prepared and forwarded to all participants by a later date.

Your Laboratory Identification Code is:

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist
(416) 235-5842